

REMARKS

Claims in the Application. Claims 1-105, 111, 113, 117, 118, 120, 121, 132-134, 140-144, 146-149, 155-159, 171-174 and 180 have been cancelled from this application. Claims 181-190 have been added herein. Claims 106, 114, 122, 130, 145, 150, 170 and 175 have been amended herein. Accordingly, Claims 106-110, 112, 114-116, 119, 122-131, 135-139, 145, 150-154, 160-168, 170, 175-179 and 181-190 are active in this application.

The claims may be grouped as follows:

- I. Claims 106-110, 112, 116, 119 and 122-129.
- II. Claims 130, 170, 114, 160, 115, 131, 135, 186, 136 and 187-189.
- III. Claims 137-139, 145 and 150-154.
- IV. Claims 161-168.
- V. Claims 181-184 and 175-179.
- VI. Claims 185 and 190.

Interview of May 4, 2009. Applicants' attorney wishes to thank Examiner Feely for the most helpful telephonic interview which was extended on May 4, 2009. At this interview, Harold D. Brannon (a co-inventor of the application), Margaret Kirick (Chief Intellectual Property Counsel for the assignee) and the undersigned attended. At the interview, the scope of the invention and the distinctions between the pending claims of the application and U.S. Patent No. 6,059,034 ("*Rickards*") were discussed, as summarized below.

In particular, Applicants discussed *Rickards*, in reference to FIG. 30, as being directed to a deformable particulate material consisting of a deformable coating 204 applied onto a non-deformable core 202. The particulate is rendered deformable and resistant to crushing by the presence of the deformable coating. In response to the comments on page 7 of the Final Office Action regarding the thickness of a deformable layer around a non-deformable particle core, Applicants indicated that they would provide data to demonstrate that a thickness of 5 microns around a non-deformable core would not render the coated particulate deformable.

Applicants argued that their invention was directed to a coating layer, penetrating layer or glazing material onto a *particulate* and not to coating of agglomerated particulates. *Rickards* discloses coating of an agglomerate to partially fill the porosity. The Examiner suggested that independent Claims 106 and 130 may be amended to specifically recite the exclusion of agglomerated particulates.

Applicants also discussed the meaning of “glazing” as recognized in the art and agreed to provide documentation to support their position.

Examiner's Rejection Over *Rickards*. The Examiner has maintained the following rejections:

- (i.) rejection of Claims 106-110, 112, 114-116, 119, 121-131, 135-140, 142, 144, 145, 150-154, 160, and 169-180 under 35 U.S.C. § 102(b) as anticipated by or, in the alternative, under 35 U.S.C. § 103(a) as obvious over *Rickards*; and
- (ii.) rejection of Claims 161-168 under 35 U.S.C. § 103(a) as being unpatentable over *Rickards*).

These grounds of rejection are respectfully traversed.

Rickards is not directed to a composite of a porous particulate material having inherent or induced permeability. On p. 10, ll. 17-23, Applicants define “inherent or induced permeability” on p. 10, ll. 17-20 as:

individual pore spaces *within the particle* ... interconnected so that fluids are capable of at least partially moving through the porous matrix, such as penetrating the porous matrix of the particle....

That Applicants consider the claimed terminology to comprise a single particle versus an agglomerate is evident by amended independent claims 106 and 130 which recite that the porous particulate material is not a cluster (or agglomerate) of particulates. In *Rickards*, an agglomerate is coated to partially fill the porosity of agglomerated particulates. *Rickards* does not disclose the use of a core particulate material which is porous, much less a core particulate material which exhibits the inherent or induced porosity of the porous particulates of the claims of Applicants. “Porosity” in *Rickards* (e.g., col. 7, l. 38; col. 8, 12; col. 17, l. 27; and col. 18, ll. 34-35) relates to the porosity of the proppant pack, porosity Claims 106 and 130 of Applicants relates to the porosity of the particulate.

Further, Claim 106 recites the ASG of the claimed selectively configured porous particulate material to be less than the ASG of the porous particulate. The ASG of the proppant pack of *Rickards* would not be less than the ASG of the deformable particulate because the deformable particulate in *Rickards* fills the porous spaces *in between* the hard non-deformable particulates. In other words, the volume of the blend of *Rickards* is substantially larger than the volume of the non-deformable proppant. The increase in volume provides an ASG to the blend which is *greater* than the ASG of the non-deformable proppant. (For the Examiner’s

consideration, Applicants attach a copy of Hall et al, Technology & Engineering, p. 286 (1983), *Attachment A*, which discusses the distinction between apparent density and ASG.]

Applicants' Claim 130 recites "the strength of the selectively configured porous particulate material is greater than the strength of the porous particulate material." The strength of the blend of *Rickards* would not be greater than the strength of the non-deformable proppant. The deformable material in the blend of *Richards* is matched to the non-deformable proppant. The strength of the blend of *Rickards* would not be different from the strength of the non-deformable proppant.

Further, a coating layer of thickness of 1 to 5 microns would not render the deformability referenced in *Rickards*. Note paragraphs 9(c), 9(d) and 9(e) of Declaration of Harold D. Brannon Under 37 CFR § 1.132, *Attachment B*. See further the conclusion of Mr. Brannon in paragraph 10.

Applicants' Claims 137, 181 and 185 recite a glazing material. The Examiner states in page 4 that any of the deformable material layers of *Rickards* "would have inherently satisfied" the glazing material. Applicants respectfully disagree. Glazing materials refer to those which are fired onto a substrate at high temperatures by firing. See, for instance, the discussion in col. 3, ll. 20-43 of U.S. Patent No. 7,250,178. Also note the instructional materials from the course *GEOL 640: Geology through Global Arts and Artifacts* at the City University of New York relating to glazing of ceramics, *Attachment C*.

Examiner's Rejection on the Ground of Double Patenting. The Examiner has rejected Claims 106-110, 112, 114-116, 119, 121-131, 135-140, 142, 144, 145, 150-154 and 160-180 on the ground of nonstatutory obviousness-type double patenting as being unpatentable over claims 95-97 of *Rickards*. This ground of rejection is also traversed.

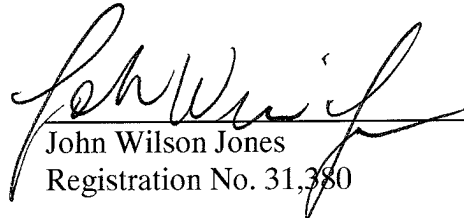
Claim 95 of *Rickards* recites a "blend of deformable particulate material and fracture proppant material, wherein at least a portion of individual particles of said deformable particulate material each comprises multiple components." As stated *supra*, Claims 106 and 130 exclude agglomerated particulates and thus the selectively configured porous particulates of Applicants are not agglomerated particulates having a coating layer. Further, Claim 106 recites the ASG of the claimed selectively configured porous particulate material to be less than the ASG of the porous particulate. The ASG of the proppant pack of *Rickards* would not be less than the ASG of the deformable particulate. In addition, the strength of the blend of *Rickards* would not be

greater than the strength of the non-deformable proppant and, as such, the double patenting rejection over Claim 130 should not stand. Applicants have also demonstrated that the claimed coating layer of thickness of 1 to 5 microns of Claim 161 would not render the deformability referenced in *Rickards*. Applicants have further demonstrated that the recitation of “glazing” in Claims 137, 181 and 185 is not taught in *Rickards*. Reconsideration of the rejection is therefore respectfully requested.

Conclusions. The Examiner is respectfully requested to telephone the undersigned should he deem it useful to expedite the prosecution of this application and issuance of a Notice of Allowance.

Respectfully submitted,

Dated: July 20, 2009

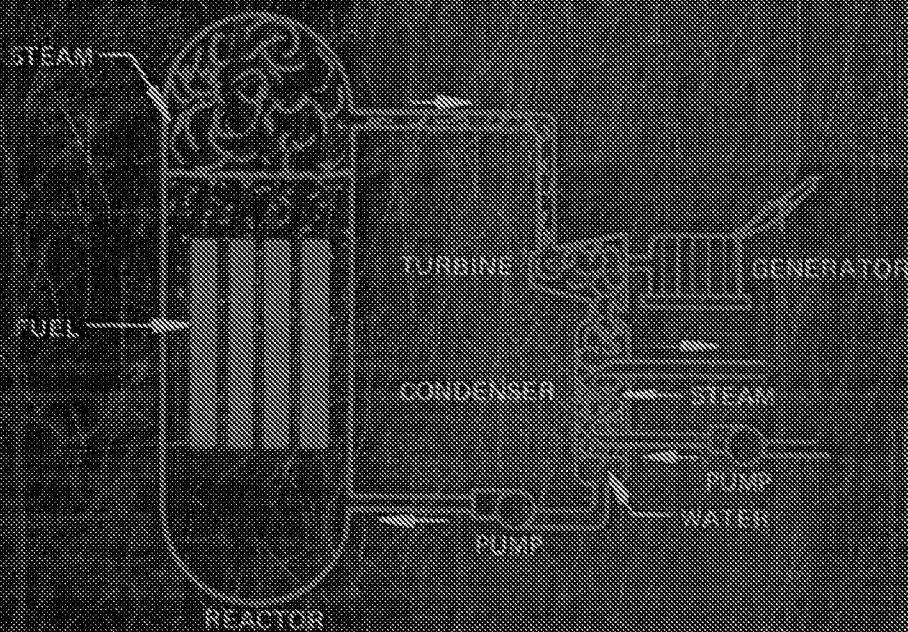


John Wilson Jones
Registration No. 31,380

Attachment A

DICTIONARY OF ENERGY

CARL W. HALL
GEORGE W. HINMAN



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$$\text{deg API} = \frac{141.5}{\text{SG } (60^{\circ}\text{F}/60^{\circ}\text{F})} - 131.5$$

APPARENT DENSITY The weight per unit volume of a material, including voids, which is a measure of the bulk of the material. Apparent density refers to the weight per unit volume whereas the apparent specific gravity denotes the specific gravity of a bulk volume, not including voids. Also see *Absolute Density*; *Bulk Density*; *Apparent Specific Gravity*.

APPARENT DIGESTIBLE ENERGY The gross energy in food consumed less the fecal energy. Also see *Fecal Energy*; *Metabolizable Energy*.

APPARENT SPECIFIC GRAVITY Denotes the specific gravity of a porous solid where the volume, *V*, excludes the permeable voids. The bulk specific gravity refers to measurements in which the volume, *V*, of the solid includes permeable and impermeable voids. Thus, bulk specific gravity is less in numerical value than apparent specific gravity. Also see *Bulk Density*; *Specific Gravity*.

AQI Air Quality Index.

AQUACULTURE (Also spelled aquiculture) The cultivation and production of plants and animals and other water-borne organisms in bodies of water, either inland or offshore. A specialized subject dealing with raising of plants and animals for human use or consumption in the large lakes and oceans is known as mariculture. Mariculture may be applied more broadly as the cultivation of marine organisms.

ARC - COAL An oil substitute made from finely ground coal developed by Atlantic Research Corporation. It is a heavy oil which will replace oils used in boilers and furnaces such as used for ships and utilities.

ARGON An inert gas making up about 1% of the atmosphere. Argon does not enter into combustion reactions. It is used as an insulator (or blanket) in nuclear reactors based on its inert qualities and heat transfer properties.

AROMATIC When referring to hydrocarbons, a hydrocarbon in which the arrangement of carbons form a closed ring or rings. The simplest aromatic hydrocarbon is benzene (C_6H_6) which results from refining petroleum and making coke from coal. The aromatic hydrocarbon is similar to the alicyclic hydrocarbon except that there are alternating bonds joining the carbon atoms. Also see *Alicyclic*; *Aliphatic*.

ASA American Standards Association.

ASAE American Society of Agricultural Engineers.

Attachment B

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Application of: Stephenson et al.	§	Examiner: Michael J. Feely
	§	
Serial No.: 10/824,217	§	Group Art 1796
	§	Unit:
	§	
Filed: April 14, 2004	§	
	§	
Title: Porous Particulate Materials	§	Attorney Docket No. 020569-03403
	§	(P202-1230B-US)
	§	

Commissioner for Patents
P.O. Box 1450
Alexandria, VA 22313-1450

DECLARATION UNDER 37 C.F.R. § 1.132 OF HAROLD DEAN BRANNON

Dear Sir:

I, Harold Dean Brannon, do hereby declare and state that:

1. I am an inventor of U.S. patent application serial no. 10/824,217 ("Application"), filed on April 14, 2004.
2. I hold a Bachelor of Science degree in Chemistry from Baylor University.
3. I am currently employed by BJ Services Company ("BJS"), in Tomball, Texas. BJS is the assignee of Application. I am presently Senior Advisor, Fracturing, at BJS where I am responsible for management and support of company fracturing technologies and represent the company in external technical activities. I have been working at BJS for 18 years.
4. Prior to my employment with BJS, I worked for 10 years in research and technical management positions for Dowell Schlumberger where I earned several awards.
5. I have a total of over 28 years industrial research and development experience, including experience in research management. I am an industry recognized expert in areas of fracturing treatment, applications, processes, and products, fracturing treatment design and evaluation, fracturing fluid systems, breakers, proppant transport, fracture conductivity, propping agents, proppant flowback control, polymeric damage

remediation, and continuous mixing process. I have experience in the pumping services business, having held positions of responsibility in areas such as research and development, technical support, field engineering, applied research, chemical and equipment manufacturing, marketing and applied technology management.

6. I have authored over 70 technical publications and papers. Not only am I a member of the Society of Petroleum Engineers ("SPE"), but I am also the current Associate Editor of the SPE Production & Operations Journal. I have served on several SPE Technical Publication Committees, including serving as Chair of the 2003 ATCE Well Stimulation Committee. I also serve as an advisor to industry funded research and development projects for DOE, API, CEA and several university JIPs.

7. I am an inventor of the following U.S. Patents:

U.S. Patent No. 7,528,096 entitled Structured Composite Compositions for Treatment of Subterranean Wells;

U.S. Patent No. 7,494,711 entitled Coated Plastic Beads and Methods of Using Same to Treat a Wellbore or Subterranean Formation;

U.S. Patent No. 7,472,751 entitled Method of Treating Subterranean Formations Using Mixed Density Proppants or Sequential Proppant Stages;

U.S. Patent No. 7,426,961 entitled Method of Treating Subterranean Formations with Porous Particulate Materials;

U.S. Patent No. 7,322,411 entitled Method of Stimulating Oil and Gas Wells Using Deformable Proppants;

U.S. Patent No. 7,213,651 entitled Methods and Compositions for Introducing Conductive Channels into a Hydraulic Fracturing Treatment;

U.S. Patent No. 7,210,528 entitled Method of Treatment Subterranean Formations Using Multiple Proppant Stages or Mixed Proppants;

U.S. Patent No. 7,207,386 entitled Method of Hydraulic Fracturing to Reduce Unwanted Water Production;

U.S. Patent No. 6,749,025 entitled Lightweight Methods and Compositions for Sand Control;

U.S. Patent No. 6,508,305 entitled Compositions and Methods for Cementing Using Elastic Particles;

U.S. Patent No. 6,364,018 entitled Lightweight Methods and Compositions for Well Treating;

U.S. Patent No. 6,330,916 entitled Formation Treatment Method Using Deformable Particles;

U.S. Patent No. 6,302,209 entitled Surfactant Compositions and Uses Therefor;

U.S. Patent No. 6,059,034 entitled Formation Treatment Method Using Deformable Particles;

U.S. Patent No. 5,881,813 entitled Method for Improved Stimulation Treatment;

U.S. Patent No. 5,547,026 entitled Crosslinked Guar Based Blocking Gel System for Use at Low to High Temperatures;

U.S. Patent No. 5,247,995 entitled Method of Dissolving Organic Filter Cake Obtained From Polysaccharide Based Fluids Used in Production Operations and Completions of Oil and Gas Wells;

U.S. Patent No. 5,103,905 entitled Method of Optimizing the Conductivity of a Propped Fractured Formation;

U.S. Patent No. 4,828,034 entitled Method of Hydrating Oil Based Fracturing Concentrate and Continuous Fracturing Process Using Same;

U.S. Patent No. 4,801,389 entitled High Temperature Guar-Based Fracturing Fluid.

8. I am the author of the following Technical Publications/Presentations:

Asadi, M., Penny, G.S., van der Bas, F., Brannon, H.D., Hawkins, G.W., Lord, D.L., McElfresh, P.M., Tayler, D.M. , “A New Industry Standard To Measure Rheology of Completion Fluids”, 2006, SPE No. 98094-MS;

Brannon, H.D., Wood, W.D., Wheeler, R.S., “Large-Scale Laboratory Investigation of the Effects of Proppant and Fracturing-Fluid Properties on Transport”, 2006, SPE No. 98005-MS;

Brannon, H., Wood, B., Wheeler, R., “The Quest for Improved Proppant Placement: Investigation of the Effects of Proppant Slurry Component Properties on Transport”, 2005, SPE No. 95675-MS;

Brannon, H.D., Starks II, T.R., "Maximizing Return-On-Fracturing-Investment by Using Ultra-Lightweight Proppants to Optimize Effective Fracture Area: Can Less Be More?", 2009, SPE No. 119385-MS;

Brannon, H.D., Stephenson, C.J., Freeman, E.R., Anschutz, D.A., Renkes, J.J., Rickards, A.R., "Critical Review of Industry-Standard Specifications for Frac Sand and Implications on Source Qualification for Use in Today's Typical Fracturing Applications", 2008, SPE No. 116089-MS;

Brannon, H.D., Starks II, T.R., "The Impact of Effective Fracture Area and Conductivity on Fracture Deliverability and Stimulation Value", 2008, SPE No. 116057-MS;

Brannon, H.D., Stephenson, C.J., Freeman, E.R., Anschutz, D.A., Renkes, J.J., Rickards, A.R., "Using Industry Standards As a Way to Predict Sand Performance and Approve Sand Deposits: Is There a Catch 22?", 2008, SPE No. 116054-MS;

Berry, Sandra L., Boles, Joel L., Brannon, Harold D., Beall, Brian B., "Performance Evaluation of Ionic Liquids as a Clay Stabilizer and Shale Inhibitor", 2008, SPE No. 112540-MS;

Kaufman, P.B., Anderson, R.W., Ziegler, M., Neves, A.R., Parker, M.A., Abney, K., de Paiva Cortes, G.W.K., Joyce, S., Penny, G.S., "Introducing New API/ISO Procedures for Proppant Testing", 2007, SPE No. 110697-MS;

Asadi, M., Penny, G.S., Ainley, B.R., Archacki, D.J., Bas van der, F., Bern, P.A., Brannon, H.D., Cobianco, S., Ghalambor, A., McElfresh, P.M., Milton-Tayler, D., Parker, M.A., "A New Technical Standard Procedure To Measure Stimulation and Gravel-Pack Fluid Leakoff Under Static Conditions", 2007, SPE No. 106243-MS;

Brannon, H.D., Wood, W.D., Wheeler, R.S., "Improved Understanding of Proppant Transport Yields New Insight to the Design and Placement of Fracturing Treatments", 2006, SPE No. 102758-MS;

Brannon, H.D., Malone, M.R., Rickards, A.R., Wood, W.D., Edgeman, R.J., Bryant, J.L., "Maximizing Fracture Conductivity with Proppant Partial Monolayers: Theoretical Curiosity or Highly Productive Reality?", 2004, SPE No. 90698-MS;

Wood, W.D., Brannon, H.D., Rickards, A.R., Stephenson, C., “Ultra-Lightweight Proppant Development Yields Exciting New Opportunities in Hydraulic Fracturing Design”, 2003, SPE No. 84309-MS;

Rickards, A.R., Brannon, H.R., Wood, W.D., Stephenson, C.J., “High Strength, Ultra-Lightweight Proppant Lends New Dimensions to Hydraulic Fracturing Applications”, 2003, SPE No. 84308-MS;

Stephenson, C.J., Rickards, A.R., Brannon, H.D., “Is Ottawa Still Evolving?”, API Specifications and Conductivity, 2003, SPE No. 84304-MS;

Brannon, H.D., Tjon-Joe-Pin, R.M., Carman, P.S., Wood, W.D., “Enzyme Breaker Technologies: A Decade of Improved Well Stimulation”, 2003, SPE No. 84213-MS.

Brannon, H.D., Rickards, A.R., Stephenson, C.J., “Exceptional Proppant Flowback Control for the Most Extreme Well Environments: The Shape of Things to Come”, 2003, SPE No. 80509-MS;

Wood, W.D., Brannon, H.D., Ault, M.G., Matson, R.P., Carman, P.S., “A Case Study of Long-Term Production Enhancement Derived from Usage of Organo-Borate Crosslinked Fracturing Fluids”, 2002, SPE No. 77747-MS;

Stephenson, C., Ward, B., Taylor, D.M., Brannon, H.D., Rickards, A., “Exceptional Proppant Flowback Control for the Most Extreme Well Environments: The Shape of Things to Come”, 2002, SPE No. 77681-MS;

Stephenson, C.J., Rickards, A.R., Brannon, H.D., “Increased Resistance to Proppant Flowback by Adding Deformable Particles to Proppant Packs Tested in the Laboratory”, 1999, SPE No. 56593-MS;

Rickards, A., Lacy, L., Brannon, H., Stephenson, C., Bilden, D., “Need Stress Relief? A New Approach to Reducing Stress Cycling Induced Proppant Pack Failure”, 1998, SPE No. 49247-MS;

Beall, B.B., Tjon-Joe-Pin, R., Brannon, H.D., “Progressive Technology Increases Flow Capacity in Horizontal Wells”, 1997, SPE No. 39230-MS;

Beall, B.B., Tjon-Joe-Pin, R., Brannon, H.D., “Field Experience Validates Effectiveness of Drill-In Fluid Cleanup System”, 1997, SPE No. 38570-MS;

Beall, B.B., Tjon-Joe-Pin, R.M., Brannon, H.D., "New Treatment Improved Cleanup of Horizontal and/or Openhole Completions: A Case History", 1997, SPE No. 38327-MS;

Tjon-Joe-Pin, R.M., Brannon, H.D., Beall, B.B., "Biotechnological Treatment Removes Xanthan-Based Skin Damage", 1997, SPE No. 38304-MS;

Brannon, H.D., Pulsinelli, R.J., "Breaker Concentrations Required to Improve the Permeability of Proppant Packs Damaged by Concentrated Linear and Borate-Crosslinked Fracturing Fluids", 1992, SPE No. 21583-PA;

Brannon, H.D., Pulsinelli, R.J., "Breaker Concentrations Required to Improve the Permeability of Proppant Packs Damaged by Concentrated Linear and Borate-Crosslinked Fracturing Fluids", 1990, SPE No. 20135-MS;

Gulbis, J., King, M.T., Hawkins, G.W., Brannon, H.D., "Encapsulated Breaker for Aqueous Polymeric Fluids", 1992, SPE No. 19433-PA;

Brannon, H.D., Pulsinelli, R.J., "Evaluation of the Breaker Concentrations Required to Improve the Permeability of Proppant Packs Damaged by Hydraulic Fracturing Fluids", 1990, SPE No. 19402-MS;

9. To establish the unexpected results obtained in the present application and as set forth in the claims, the following experiments were conducted under my supervision and control:

a.) An ultra lightweight proppant (Specimen 1) was prepared by applying a coating/penetrating layer onto a porous ceramic. The particle size of the porous ceramic was 20/40 mesh. The coating was 2% by weight of particle epoxy inner coating/penetrating material followed by 2% by weight of particle phenol formaldehyde resin outer coating material. The proppant is described as "porous ceramic -- 2/2" in Example 1 of the instant application. SEM photographs showed a coating thickness of approximately 5 microns. *See* FIG. 1. A micrograph of a thin section of Specimen 1 is further set forth in FIG. 2.

b.) Particle hardness testing was then conducted on Specimen 1 and Specimen 2. Specimen 2 was a deformable particulate and consisted of a 20/40 mesh ground or crushed nut shells coated with protective or hardening coating as described in Example 4 as "III." of the instant application. Particle hardness testing was conducted using an

Instron Model 3345 with a 1kN load cell and Series IX software to assess the displacement (or change in particle width) observed when a load is applied to a single particle.

c.) The response to stress loading on particles of Specimen 1 and Specimen 2 are shown in FIG. 3. As load was increased on Specimen 2 (Y-axis), particle width decreased throughout the load application range (0 – 48 lbf). Such behavior is indicative of deformable material character. Conversely for Specimen 1 at ~5 lbf, an abrupt width change as particle failure (or crush) was observed. Such behavior is representative of non-deformable particle character.

d.) Particle strength testing was conducted on 10 individual particles of Specimen 1 and Specimen 2 to estimate the properties of a bulk specimen. The original particle size for each of the specimens was 0.03 inches (20/40 mesh having a median diameter of approximately 25 mesh). As illustrated in FIG. 4, 10 particles of Specimen 2 were tested, labeled as Specimens 1-10. The range of both axis scales is much larger due to the significantly higher force and displacement values measured. Each of the 10 specimens was observed to experience increasing displacement with application of increasing force (or stress). Note that the displacement of Specimen 2 was about 0.02 inches at the maximum applied load or 65% (0.02/0.03) of the original width, without failing. This phenomena illustrates the deformative character of the particulate materials of Specimen 2.

e.) Particle strength testing was conducted on 5 to 10 individual particles of Specimen 1 to estimate the properties of a bulk specimen. As shown in FIG. 5, 10 particles of Specimen 1 were tested, labeled as Specimens 1-10. The range of both axis scales is much smaller than those set forth in FIG. 4. Each of the 10 specimens was observed to suffer abrupt failure at between 1 - 12 lbf., with the associated 0.0005 -0.004 in displacement measured at the point of failure. For instance, in contrast to the specimens of Specimen 2 referenced in the paragraph above, particle failure occurred at 0.0005 – 0.004 inches displacement, or approximately 3% (0.001/0.03) of the unstressed diameter.

10. I am a co-inventor of U.S. Patent No. 6,059,034 (“*Rickards*”). The deformability referenced in *Rickards* would not exist within the selectively configured

porous particulate material set forth in Claim 161 of the instant application as amended by the amendment filed concurrently with this Declaration. In fact, the thickness of the deformable materials of *Rickards* would have to be a minimum of about 100 microns in order to impart deformability to the non-deformable material of *Rickards*. The thickness of a 20/40 mesh of non-deformable proppant is typically about 600 microns. A thickness of 1 to 5 microns would be insufficient to impart deformability to the non-deformable proppant of *Rickards*.

I hereby declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under 18 U.S.C. 1001 and that such willful false statements may jeopardize the validity of the application or any patent issued thereon.

DATED: June 17, 2009
June 17, 2009

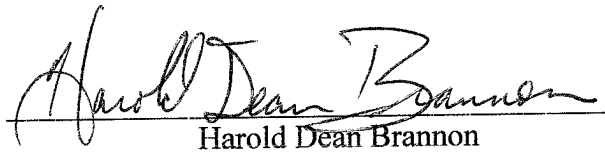

Harold Dean Brannon

FIG. 1

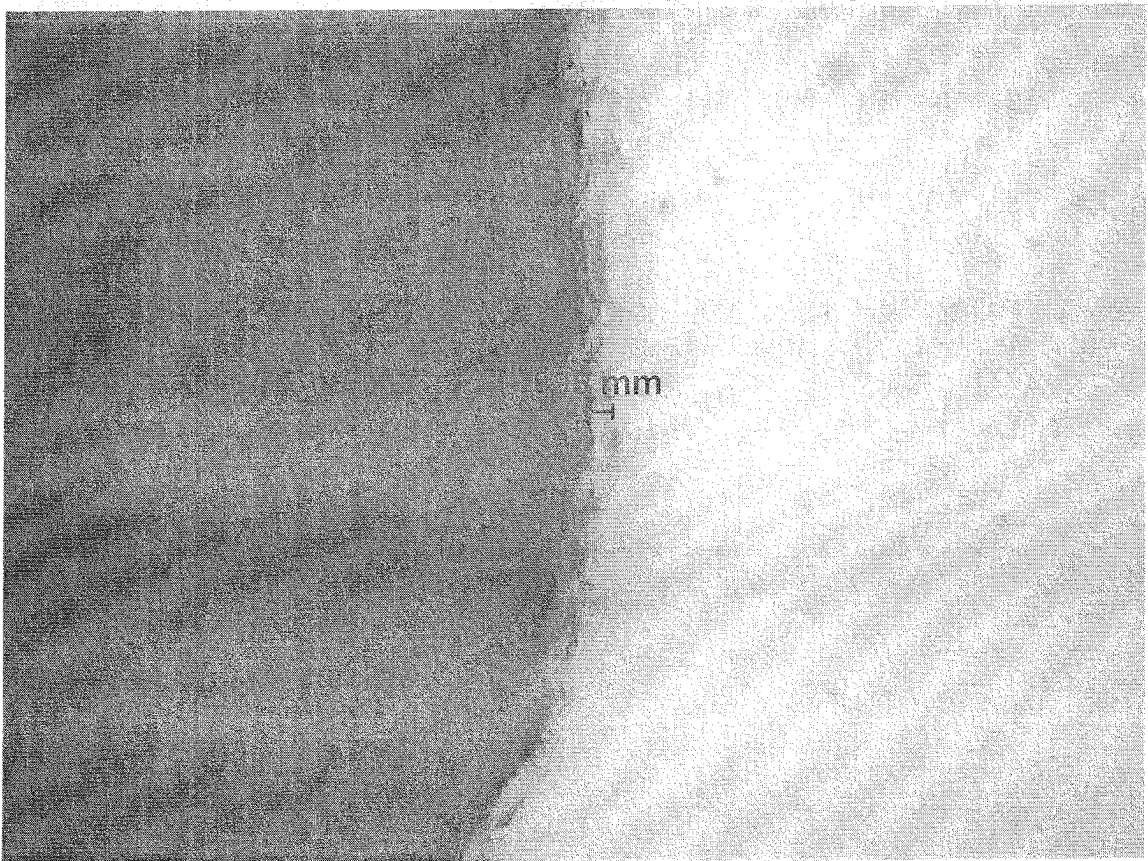
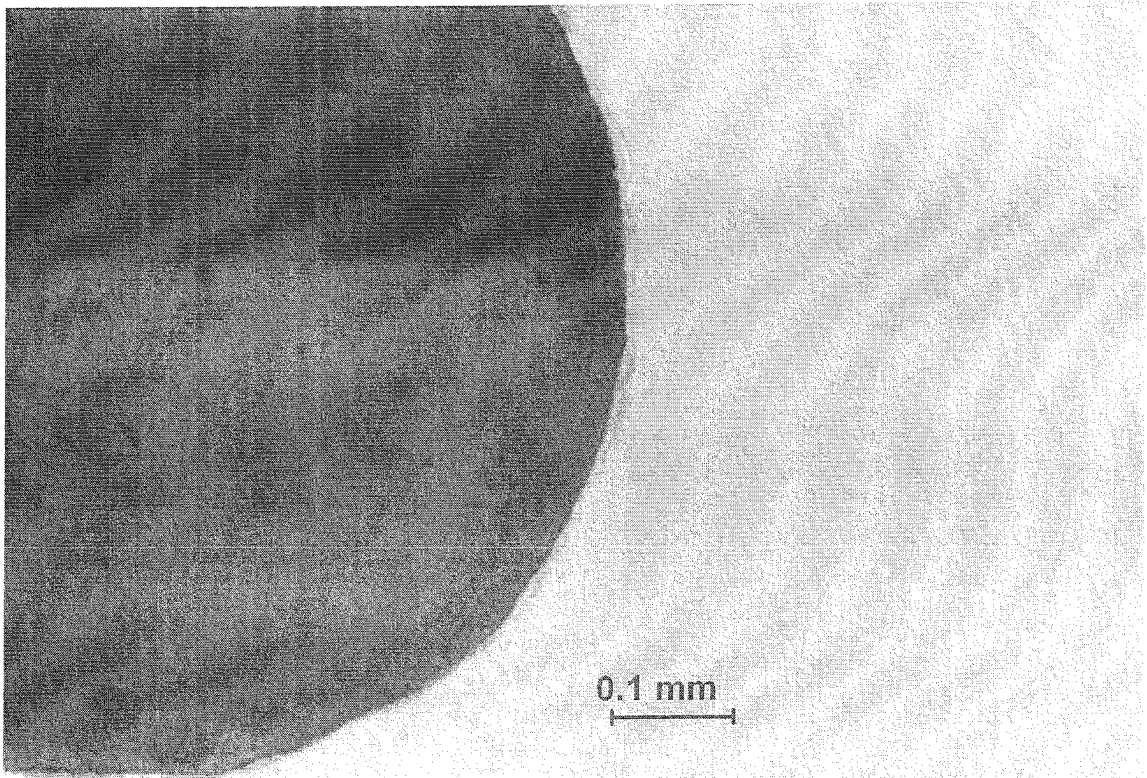


FIG. 2

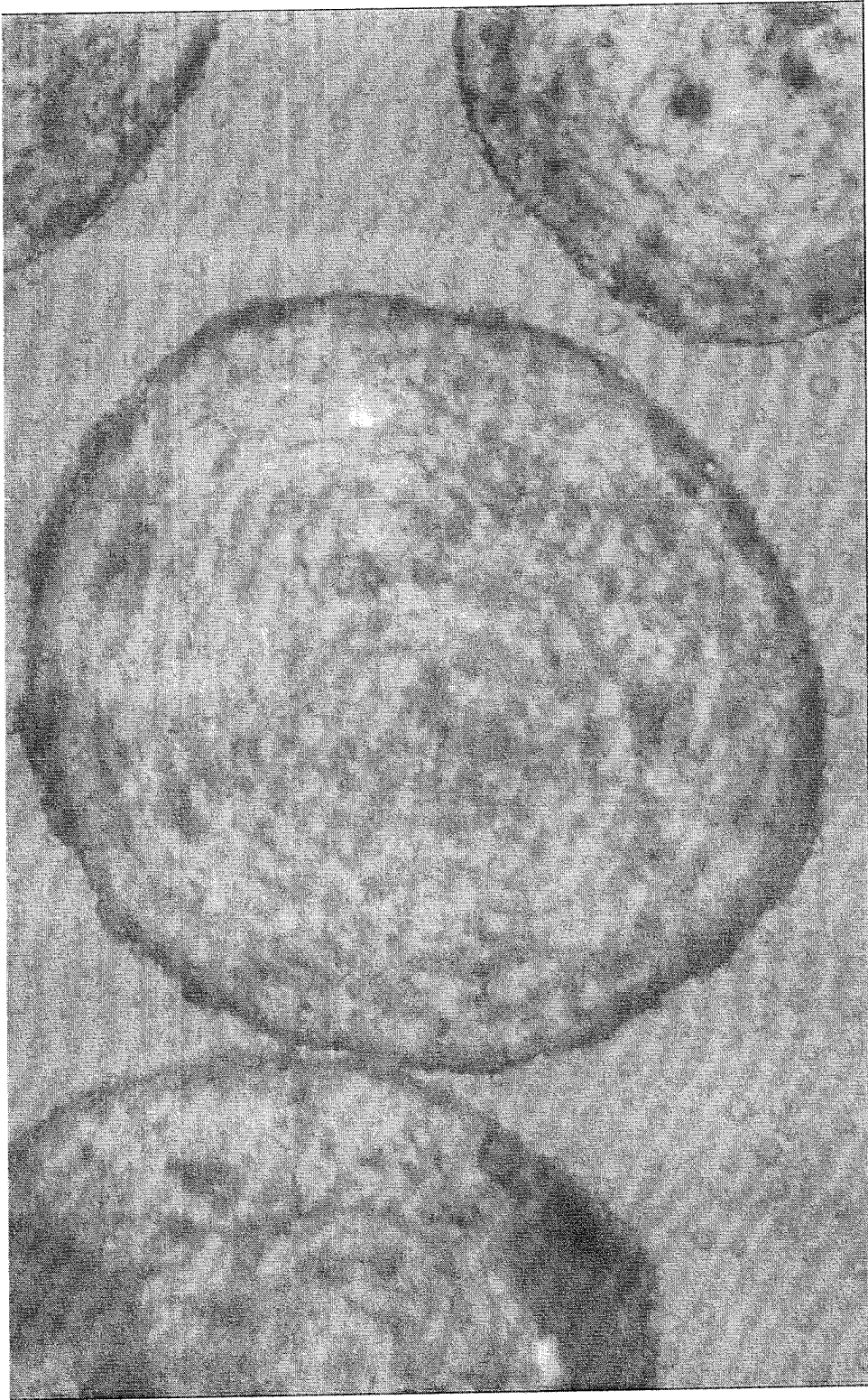


FIG. 3

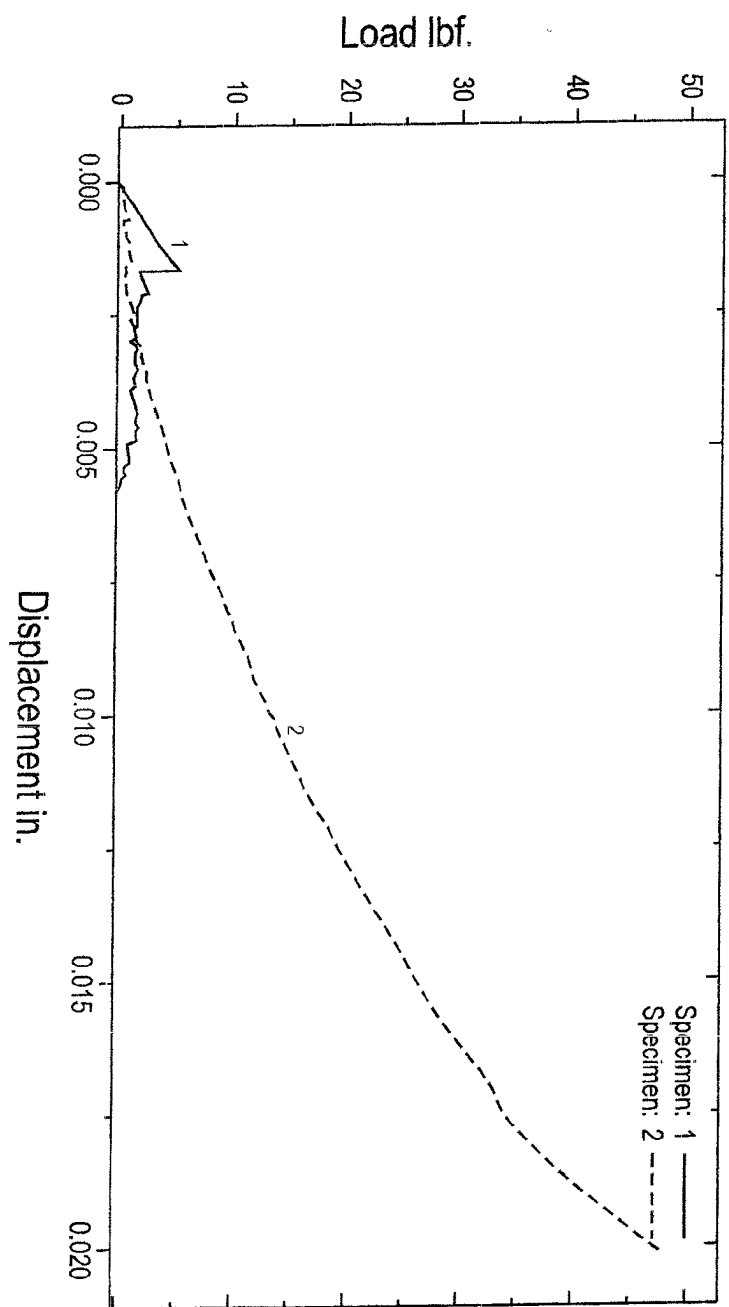
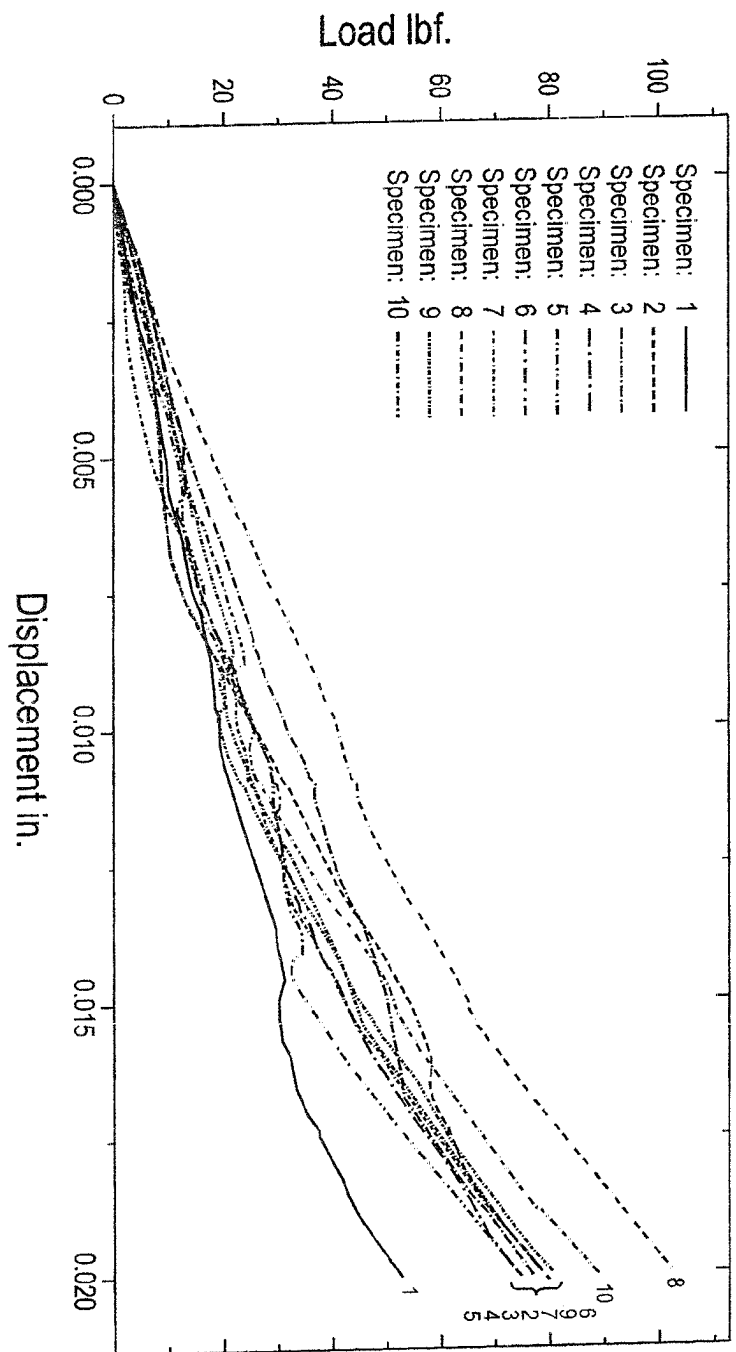


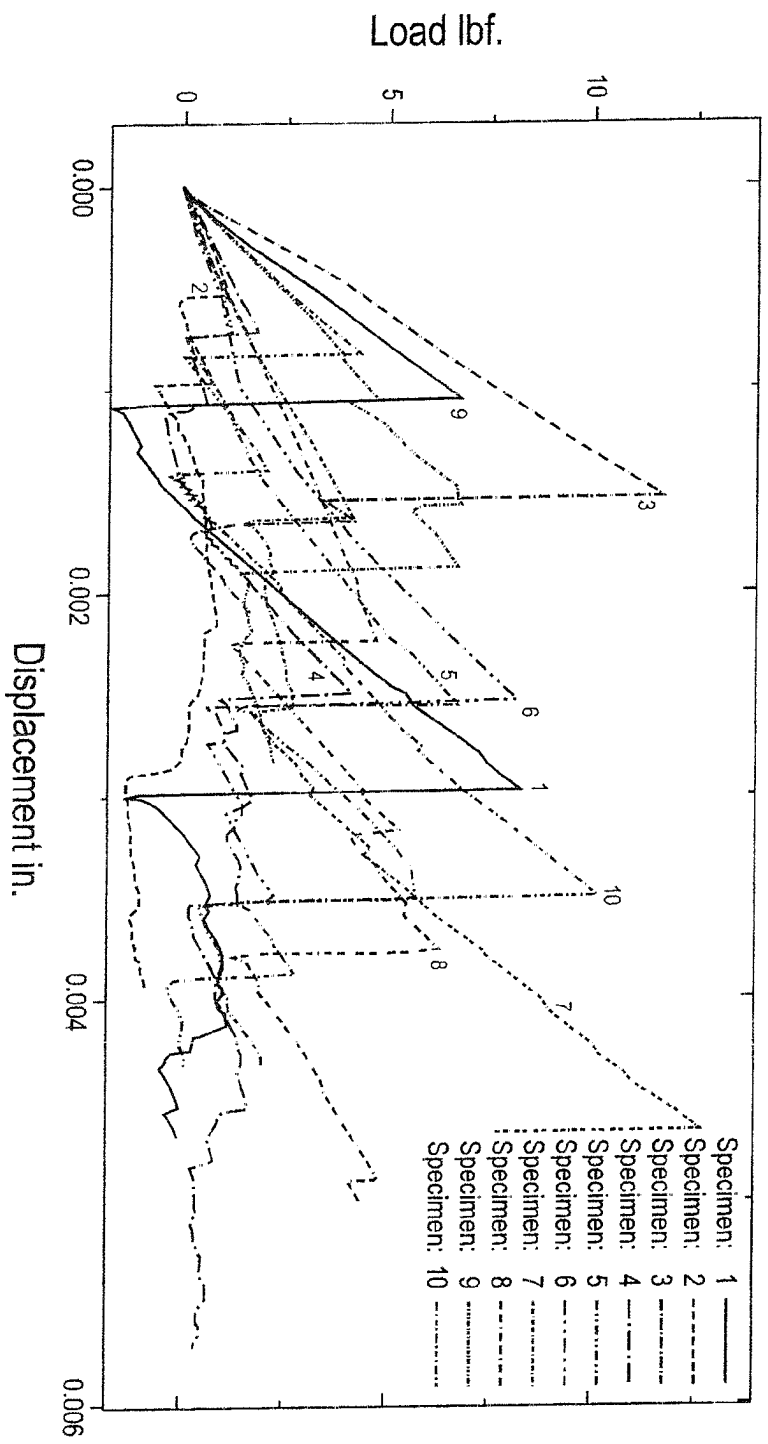
FIG. 4



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FIG. 5



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Attachment C

A glaze is a waterproof silica glass on the surface of a ceramic pot, and was first produced by the Mesopotemians around 2000 BC. (A clay tablet dated at 1700BC from northern Iraq records a recipe for a green glaze (Cooper, 1988)). A glaze is produced by melting a mixture that consists mostly of powdered minerals, although organic materials such as wood-ash and bone-ash may also be added. Each ingredient in a glaze mix serves one or more of 4 essential purposes:

Colorant: Various metals (e.g., Fe, Co, Cu, Mn, Cr, Ti, Pb).

[illegible]

To create an effective glaze, a potter must mix the components in just the right proportions; enough flux to make the silica melt but not too much to make it melt too early and become runny; enough amphoteric to make the glaze stick to the pot but not too much to make the glaze thick and "globby"; the right balance of metals to get the desired color shade. This ideal mixture must also be ideally suited for the temperature to which the kiln is fired.

With so many variables it is difficult to fully predict the results of a given recipe. Accordingly, potters adopt an experimental approach, similar to that of an experimental petrologist; a potter will systematically mix different proportions of critical components, apply them to test pieces of pottery, and fire them to the desired temperature. When a test piece has been fired the potter can select the recipe that created the most desired result. Having narrowed down the range of composition that produces desirable results, the potter may run additional experiments to hone the recipe. (What if I added 4% calcite instead of 5%? What if I added a half percent of cobalt oxide with the 2% malachite that I tried last time?)

Note that this approach is scientific experimentation and hypothesis testing. For example, in asking "What if I added 6% calcite instead of 5%?" the potter is interested in testing a hypothesis that increasing the flux by 20% will reduce the melting point further, and produce a more glassy product. In asking "What if I added a half percent of cobalt oxide with the 2% malachite that I tried last time?", the potter is interesting in testing his hypothesis that a rich blue-green color would result by mixing the two transition metal-bearing colorants.

The following table lists minerals that are used commonly in glazes. For each of the minerals determine how it would contribute to a glaze recipe

Mineral	Formula	Glass Maker?	Flux?	Amphoteric?	Colorant?
Albite	$\text{NaAlSi}_3\text{O}_8$				
Calcite	CaCO_3				
Cassiterite	SnO_2				
Dolomite	$\text{CaMg}[\text{CO}_3]_2$				
Flint/Quartz	SiO_2				
Hematite	Fe_2O_3				
Kaolinite	$\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$				
Malachite	$\text{Cu}_2(\text{CO}_3)(\text{OH})_2$				
Microcline	KAlSi_3O_8				
Pyrolusite	MnO_2				
Rutile	TiO_2				
Wollastonite	CaSiO_3				
Zincite	ZnO				

List four pairings of minerals that would potentially produce a workable glaze (not considering colorants)?

List four sets of three minerals that would potentially produce a workable glaze?

SILICATE GLAZES and MAGMAS

Glaze recipes are composed primarily of silicate minerals, including quartz and feldspars which are the most abundant minerals in most igneous rocks. The presence of lesser amounts of iron-bearing minerals in igneous rocks is what controls their color (Fe is the most abundant transition metal in the crust), just as the addition of small amounts of a transition-metal-bearing mineral provides colorant to a glaze recipe. Potters have experimented with glazes to perfect their recipes—glazes that have the right color and which melt at the temperatures that are reached within their kilns, and which form colored glass when they are subsequently cooled relatively quickly. Similarly, geologists have experimented with igneous rocks, but rather than simply attempting to create an eye-pleasing glass, geologists have sought to understand how rocks melt, and under what conditions, and how igneous rocks form within the Earth.

Beginning with the pioneering work of Norman L. Bowen in 1909, geoscientists have worked to understand the genesis of magmas and igneous rocks by taking small amounts of powdered minerals of known compositions, heating them to a known temperature, holding them at that temperature until the materials reach chemical equilibrium. These heated samples were then cooled rapidly to room temperature for examination under the microscope, molten rock would freeze to a glass that held any mineral crystals that existed within the magma.

Through such methods, experimental petrologists like Bowen create phase diagrams that predict how rocks of different compositions will melt, in what order minerals within these rocks will melt, and conversely, how igneous rocks form from magmas, and in what sequence minerals will form to create a rock from magma of a given composition. The ternary phase diagram $\text{SiO}_2\text{-NaAlSi}_3\text{O}_8\text{-KAlSi}_3\text{O}_8$ (quartz-albite-K feldspar) is used to understand how granites crystallize and melt since >90% of any granite is composed of these three minerals. This ternary diagram can also be used to understand how any two mixtures of these minerals will melt.

Examine the $\text{SiO}_2\text{-NaAlSi}_3\text{O}_8\text{-KAlSi}_3\text{O}_8$ (quartz-albite-K feldspar) ternary phase diagram on the following page, and answer the following questions:

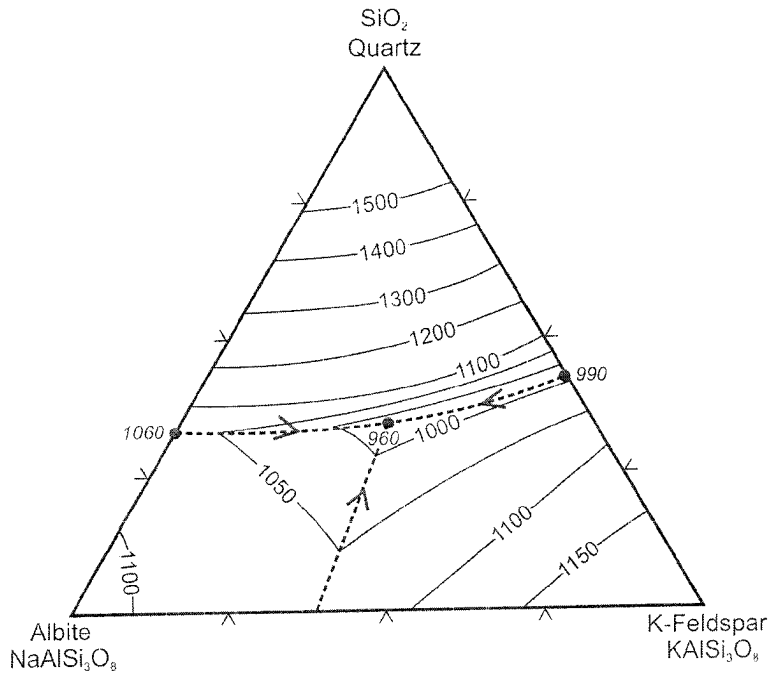
At what temperature does each of the following melt?

Quartz alone: _____ Albite alone: _____ K-Feldspar alone: _____

Quartz + Albite: _____ Quartz + K Feldspar: _____

Quartz + Albite + K-Feldspar: _____

In general, what happens to the melting temperature of an igneous rock (or glaze) when you add an additional mineral to the mix?

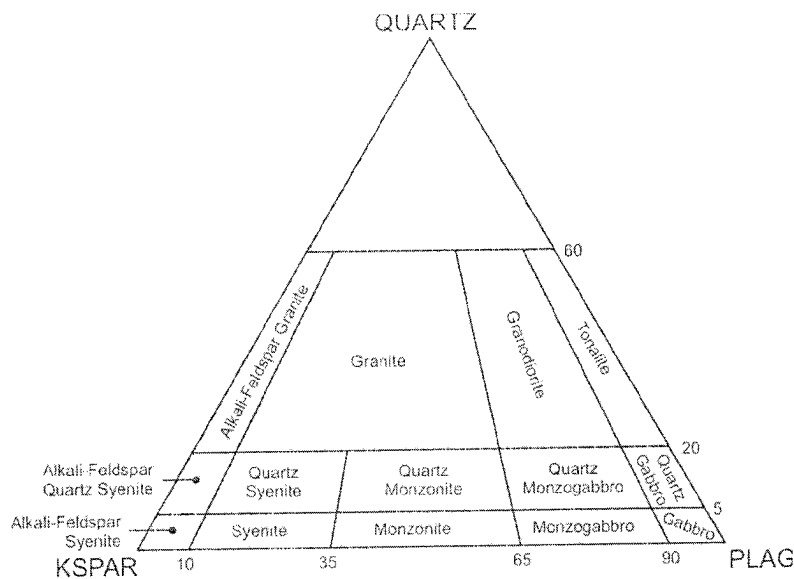


Simplified Qtz-Ab-Kfs Ternary Phase Diagram

Modified from: DEVINEAU, PICHAVANT and VILLIERAS (2005) Melting kinetics of granitic powder aggregates at 1175°C, 1 atm. European Journal of Mineralogy, v. 17, p. 387-398

Based on the Qtz-Ab-Kfs ternary phase diagram, what is the composition of rock (or recipe for glaze minerals) that will belt at the lowest temperature (eutectic)?

Quartz: _____% Albite: _____% K-Feldspar: _____%



International Union of Geological Sciences (IUGS) Classification Scheme for Intrusive Igneous Rocks

Based on the IUGS classification chart for intrusive igneous rocks, what rock forms from the lowest temperature (eutectic) melt in the Qtz-Ab-Kfs system? (Note that albite is a sodic plagioclase.)

Examine hand samples of the minerals that you will be using in preparing a glaze experiment, and document/review their physical properties.

Quartz: SiO_2

Hardness: _____

Color: _____

Cleavage/Fracture: _____

Streak: _____

Quartz, var. Flint: SiO_2

Hardness: _____

Color: _____

Cleavage/Fracture: _____

Streak: _____

Albite: $\text{NaAlSi}_3\text{O}_8$

Hardness: _____

Color: _____

Cleavage/Fracture: _____

Streak: _____

Microcline: KAlSi_3O_8

Hardness: _____

Color: _____

Cleavage/Fracture: _____

Streak: _____

Calcite: CaCO_3

Hardness: _____

Color: _____

Cleavage/Fracture: _____

Streak: _____

Hematite: Fe_2O_3

Hardness: _____

Color: _____

Cleavage/Fracture: _____

Streak: _____

Malachite: $\text{Cu}_2(\text{CO}_3)(\text{OH})_2$

Hardness: _____

Color: _____

Cleavage/Fracture: _____

Streak: _____

Pyrolusite: MnO_2

Hardness: _____

Color: _____

Cleavage/Fracture: _____

Streak: _____

Break into groups, as assigned by your instructor and prepare two experimental test tiles to investigate the melting temperature of mineral mixtures. Each group will create two test tile (one rectangular tile with five wells; one triangular tile with 10 wells), as assigned in the table below.

- Fill each well according to the recipes on the following worksheet. For example, on the Quartz-Albite tile the 3-1 well would be filled with 3 parts quartz and 1 part albite, whereas the 1-3 well would be 1 part quartz and 3 parts albite. Use the smallest measuring spoon provided (1/8 teaspoons / smidgeon), leveling off the measurements with a popsicle stick.
- For Groups 3 through 6, choose a colorant/flux from the table on Page 2, and add 1 part of your chosen colorant/flux to each well. Add the name of the mineral that you chose to the table below by filling in the blanks.
- When all of the mineral powders have been added to each well on the test tiles, add approximately 10 drops of water to each well, and mix each with a popsicle stick to create a well mixed runny paste. The water will absorb into the bisqued tile very quickly.
- Label your tiles with your group number and minerals used by applying an iron oxide-based wash with a fine paintbrush

Group 1	Group 2	Group 3	Group 4	Group 5	Group 6
Qtz-Ab	Qtz-Ab	Qtz-Kfs	Qtz-Kfs	Qtz-Ab + Colorant ()	Qtz-Kfs + Flux ()
Qtz-Ab-Kfs	Qtz-Ab-Kfs	Qtz-Ab-Kfs + Colorant ()	Qtz-Ab-Kfs + Flux ()	Qtz-Ab-Kfs + Colorant ()	Qtz-Ab-Kfs + Flux ()

If the kiln is heated to ~1050°C (Pyrometric Cone 05), which mixtures in your test tiles do you predict would fully melt and form a glassy glaze? Mark a ✓ beside the corresponding wells on the following worksheet. these on the following worksheet.

If the kiln is heated to ~1100°C (Pyrometric Cone 03), which additional mixtures in your test tiles do you predict would fully melt and form a glassy glaze? Mark an ✕ beside the corresponding wells on the following worksheet.

What color(s) do you predict that your glazes will be? _____

Which group(s) do you predict will have the test tiles with the greatest number of successful glazes?

